

# THE 2000

**Mailing Label Number: EL569393835US**

2000 2001 2002 2003 2004 2005 2006 2007 2008 2009 2010 2011 2012 2013 2014 2015 2016 2017 2018 2019 2020 2021 2022 2023 2024 2025 2026 2027 2028 2029 2030 2031 2032 2033 2034 2035 2036 2037 2038 2039 2040 2041 2042 2043 2044 2045 2046 2047 2048 2049 2050 2051 2052 2053 2054 2055 2056 2057 2058 2059 2060 2061 2062 2063 2064 2065 2066 2067 2068 2069 2070 2071 2072 2073 2074 2075 2076 2077 2078 2079 2080 2081 2082 2083 2084 2085 2086 2087 2088 2089 2090 2091 2092 2093 2094 2095 2096 2097 2098 2099 2100 2101 2102 2103 2104 2105 2106 2107 2108 2109 2110 2111 2112 2113 2114 2115 2116 2117 2118 2119 2120 2121 2122 2123 2124 2125 2126 2127 2128 2129 2130 2131 2132 2133 2134 2135 2136 2137 2138 2139 2140 2141 2142 2143 2144 2145 2146 2147 2148 2149 2150 2151 2152 2153 2154 2155 2156 2157 2158 2159 2160 2161 2162 2163 2164 2165 2166 2167 2168 2169 2170 2171 2172 2173 2174 2175 2176 2177 2178 2179 2180 2181 2182 2183 2184 2185 2186 2187 2188 2189 2190 2191 2192 2193 2194 2195 2196 2197 2198 2199 2200 2201 2202 2203 2204 2205 2206 2207 2208 2209 2210 2211 2212 2213 2214 2215 2216 2217 2218 2219 2220 2221 2222 2223 2224 2225 2226 2227 2228 2229 2230 2231 2232 2233 2234 2235 2236 2237 2238 2239 2240 2241 2242 2243 2244 2245 2246 2247 2248 2249 2250 2251 2252 2253 2254 2255 2256 2257 2258 2259 2260 2261 2262 2263 2264 2265 2266 2267 2268 2269 2270 2271 2272 2273 2274 2275 2276 2277 2278 2279 2280 2281 2282 2283 2284 2285 2286 2287 2288 2289 2290 2291 2292 2293 2294 2295 2296 2297 2298 2299 2300 2301 2302 2303 2304 2305 2306 2307 2308 2309 2310 2311 2312 2313 2314 2315 2316 2317 2318 2319 2320 2321 2322 2323 2324 2325 2326 2327 2328 2329 2330 2331 2332 2333 2334 2335 2336 2337 2338 2339 2340 2341 2342 2343 2344 2345 2346 2347 2348 2349 2350 2351 2352 2353 2354 2355 2356 2357 2358 2359 2360 2361 2362 2363 2364 2365 2366 2367 2368 2369 2370 2371 2372 2373 2374 2375 2376 2377 2378 2379 2380 2381 2382 2383 2384 2385 2386 2387 2388 2389 2390 2391 2392 2393 2394 2395 2396 2397 2398 2399 2400 2401 2402 2403 2404 2405 2406 2407 2408 2409 2410 2411 2412 2413 2414 2415 2416 2417 2418 2419 2420 2421 2422 2423 2424 2425 2426 2427 2428 2429 2430 2431 2432 2433 2434 2435 2436 2437 2438 2439 2440 2441 2442 2443 2444 2445 2446 2447 2448 2449 2450 2451 2452 2453 2454 2455 2456 2457 2458 2459 2460 2461 2462 2463 2464 2465 2466 2467 2468 2469 2470 2471 2472 2473 2474 2475 2476 2477 2478 2479 2480 2481 2482 2483 2484 2485 2486 2487 2488 2489 2490 2491 2492 2493 2494 2495 2496 2497 2498 2499 2500 2501 2502 2503 2504 2505 2506 2507 2508 2509 2510 2511 2512 2513 2514 2515 2516 2517 2518 2519 2520 2521 2522 2523 2524 2525 2526 2527 2528 2529 2530 2531 2532 2533 2534 2535 2536 2537 2538 2539 2540 2541 2542 2543 2544 2545 2546 2547 2548 2549 2550 2551 2552 2553 2554 2555 2556 2557 2558 2559 2560 2561 2562 2563 2564 2565 2566 2567 2568 2569 2570 2571 2572 2573 2574 2575 2576 2577 2578 2579 2580 2581 2582 2583 2584 2585 2586 2587 2588 2589 2590 2591 2592 2593 2594 2595 2596 2597 2598 2599 2600 2601 2602 2603 2604 2605 2606 2607 2608 2609 2610 2611 2612 2613 2614 2615 2616 2617 2618 2619 2620 2621 2622 2623 2624 2625 2626 2627 2628 2629 2630 2631 2632 2633 2634 2635 2636 2637 2638 2639 2640 2641 2642 2643 2644 2645 2646 2647 2648 2649 2650 2651 2652 2653 2654 2655 2656 2657 2658 2659 2660 2661 2662 2663 2664 2665 2666 2667 2668 2669 2670 2671 2672 2673 2674 2675 2676 2677 2678 2679 2680 2681 2682 2683 2684 2685 2686 2687 2688 2689 2690 2691 2692 2693 2694 2695 2696 2697 2698 2699 2700 2701 2702 2703 2704 2705 2706 2707 2708 2709 2710 2711 2712 2713 2714 2715 2716 2717 2718 2719 2720 2721 2722 2723 2724 2725 2726 2727 2728 2729 2730 2731 2732 2733 2734 2735 2736 2737 2738 2739 2740 2741 2742 2743 2744 2745 2746 2747 2748 2749 2750 2751 2752 2753 2754 2755 2756 2757 2758 2759 2760 2761 2762 2763 2764 2765 2766 2767 2768 2769 2770 2771 2772 2773 2774 2775 2776 2777 2778 2779 2780 2781 2782 2783 2784 2785 2786 2787 2788 2789 2790 2791 2792 2793 2794 2795 2796 2797 2798 2799 2800 2801 2802 2803 2804 2805 2806 2807 2808 2809 2810 2811 2812 2813 2814 2815 2816 2817 2818

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COMPOSITIONS FOR CONTROLLED RELEASE

## 10

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The application of coating materials on various substrates is a well known technique for controlling or delaying the release of that substrate when

5     deteriorate. For example, the encapsulation of an oil well chemical intended to be  
functional in a subterranean oil recovery operation insulates the chemical from  
immediate contact with the environment into which it is introduced, thereby  
maintaining the integrity of the chemical and preventing undesirable  
agglomeration or reaction until the chemical may be released in the desired  
10    location.

      A variety of chemical compositions are conventionally used as controlled  
release agents in the food, cosmetic, paint, pharmaceutical, personal care,  
household, and polymer and oil field industries. Conventional controlled release  
compositions typically operate as encapsulating agents and include gum arabic,  
15    dextrins, low viscosity modified starches, arabinogalactan, gum acacia, casein,  
gelatin, carboxymethyl cellulose, tragacanth, karaya, sodium alginate, tannin, and  
celluloses.

      Solubilized starches having been disclosed as agents for use in oil field  
encapsulation technology. US Patent No. 4,704,214, for example, describes the  
20    use of polysaccharides and polyols to form matrices for encapsulating various  
ingredients, especially oil field chemicals. In this case, a polysaccharide, which  
may be starch, is solubilized to form an inert film that surrounds an oil absorbent  
polymer. The film subsequently melts or slowly dissolves, releasing the oil-  
swelling polymer, which then closes off fractures or large pores without clogging  
25    the borehole of the oil well. The encapsulation of a wide range of oil field  
chemicals is also discussed in U.S. Patent No. 5,922,652 wherein a complex  
colloid, which may be a gelatinized starch, forms a coacervate with water-  
immiscible droplets of the chemical. U.S. Patent No. 5,546,798 discloses that

5 starch may be used as a water soluble thickening agent in conjunction with a swelling clay and a sealing agent such as calcium carbonate in order to encapsulate and thus preserve oil field core sample integrity. These forms of encapsulation all require the use of solubilized or non-particulate starch in their encapsulation formulation.

10 The use of particulates, both soft and hard, are also known controlled release ingredients for use in oil field applications. As discussed in U.S. Patent Nos. 5,560,439 and 5,204,183, typical particulates are calcium carbonate, clays, urea, polyvinyl acetate powder or emulsions. Particulates are often used as fillers or swelling agents in conjunction with water soluble thickening agents such as  
15 polyethylene glycols, polyethylene glycol or a sulfonated elastomeric polymer. In particular, US Patent No. 5,628,813 describes the use of a lignosulfonate coating containing a particulate, such as talc,  $\text{TiO}_2$ , silica, clays or gypsum which functions as a filler. The lignosulfonate coating is a secondary encapsulation intended to protect a thin, water permeable polymeric coating for encapsulating  
20 pharmaceuticals, agricultural chemical and oil well chemicals. There remains a continuing need for a variety of controlled release agents that may be modified to suit a number of environments, particularly oil field applications.

Accordingly, it has now been discovered that particulate starch desirably adsorbs chemicals, thereby providing stable, controlled release chemical  
25 compositions suitable for oil field applications.

The present invention relates to chemical compositions in which the chemical, particularly at least one oil well chemical, is adsorbed onto particulate starch. The particulate starch of the present invention includes granular starch and, resistant starch made therefrom, particularly granular starch.

10           The preparation of the present chemical compositions comprises mixing the chemical with the particulate starch. Adjuncts useful in controlled release formulations may optionally be added to the mixture. The mixture may also be optionally subjected to further treatment so that a stable, free-flowing chemical composition is obtained.

15           The chemical compositions of this invention are advantageously biodegradable and exhibit good controlled release properties, particularly in oil well applications.

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## DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to controlled release chemical compositions in which the chemical, particularly at least one oil well chemical, is adsorbed onto particulate starch. The invention further relates to the process of obtaining these compositions and their method of use.

25            Use of starch as a carrier for the oil well chemical offers many advantages  
over those carriers generally used in the industry. For example, the starch is  
biodegradable and provides a good environmental profile.

5           The term starch particulate as used herein is intended to include starches with a highly organized structure, including granular starches and resistant starches made therefrom.

10           All granular starches and flours (hereinafter "starch") may be suitable for use herein and may be derived from any native source. A native starch as used herein, is one as it is found in nature. Also suitable are starches derived from a plant obtained by standard breeding techniques including crossbreeding, translocation, inversion, transformation or any other method of gene or chromosome engineering to include variations thereof. In addition, starch derived from a plant grown from artificial mutations and variations of the above genetic composition, which may be produced by known standard methods of mutation breeding, are also suitable herein.

15           Typical sources for the starches are cereals, tubers, roots, legumes and fruits. The native source can be corn, pea, potato, sweet potato, banana, barley, wheat, rice, sago, amaranth, tapioca, arrowroot, canna, sorghum, and waxy or high amylose varieties thereof. A used herein, the term "waxy" is intended to include a starch containing at least about 95% by weight amylopectin and the term "high amylose" is intended to include a starch containing at least about 40% by weight amylose.

20           Conversion products which retain their granular structure may be derived from any of the starches, including fluidity or thin-boiling starches prepared by oxidation, enzyme conversion, acid hydrolysis, heat and or acid dextrinization, and or sheared products may also be useful herein.

5            Particularly useful are granular structures, which have been "pitted" by  
the action of enzymes or acid, leaving a still organized structure which creates a  
microporous starch. The enzymatic or acid hydrolysis of the starch granule is  
carried out using techniques well known in the art. The amount of enzyme used is  
dependent upon the enzyme, i.e., type, source and activity, as well as enzyme  
10 concentration, substrate concentration, pH, temperature, the presence or  
absence of inhibitors, and the degree and type of modification. Types of  
modifications are described herein, *infra*. These parameters may be adjusted to  
optimize the nature and extent of the "pitting" of the starch granule.

          Another particulate starch useful in the controlled release applications of  
15 the present invention is resistant starch. Resistant starch is commonly known as  
a starch not likely to be adsorbed in the small intestine of a healthy individual.  
Granular or particulate starches, such as of the RS2-type (a starch granule that  
resists digestion by pancreatic alpha-amylase) and the RS4-type (a chemically  
modified starch, such as acetylated, hydroxyalkylated, or cross-linked starch) are  
20 particularly suitable. However, resistant starches of the RS3-type (retrograded,  
nongranular starch formed by heat/moisture treatment of starch) are also suitable  
for the instant invention due to their high level of retrogradation or crystallization  
from the alignment and association of associated amylose. In the case of an  
RS3-type of starch, the order present in the original granular state has been  
25 replaced with a crystalline order associated with retrograded amylose that may  
swell a bit in water but doesn't completely solubilize.

          These types of resistant starch are well known in the art and may be  
exemplified by that disclosed in US Patent Nos. US 5,593,503 which describes a

5 method of making a granular resistant starch; US Patent Nos. 5,281,276 and  
5,409,542 which describe methods of making resistant starches of the RS3 type;  
US 5,855,946 which describes a method of making a resistant starch of the RS4-  
type; and U.S. Application Serial No. 60/157370, which describes the formation of  
a very highly resistant starch. The methods for making the resistant starches are  
10 described in the preceding references, the disclosures of which are incorporated  
herein by reference.

The starch particulate, including granular and resistant starches, may be  
modified by treatment with any reagent or combination of reagents which  
contribute to the controlled release properties of the starch, provided the  
15 modification does not destroy the particulate nature of the starch. Chemical  
modifications are intended to include crosslinked starches, including crosslinking  
the particulate starch with reactive polymers. Preferred reactive polymers include  
starches modified with aldehyde or silanol groups. Other chemical modifications  
include, without limit, acetylated and organically esterified starches,  
20 hydroxyethylated and hydroxypropylated starches, phosphorylated and  
inorganically esterified starches, cationic, anionic, nonionic, and zwitterionic  
starches, and succinate and substituted succinate derivatives of starch.  
Preferred modified starches are starch acetates having a degree of substitution  
("DS") of about up to about 1.5, particularly those disclosed in US 5,321,132,  
25 thereby improving compatibility with synthetic hydrophobic materials. Such  
modifications are known in the art, for example in *Modified Starches: Properties  
and Uses*, Ed. Wurzburg, CRC Press, Inc., Florida (1986). Another particularly  
suitable modified starch is starch octenylsuccinate, aluminum salt.

5           Other suitable modifications and methods for producing particulate  
starches are known in the art and disclosed in U.S. Patent No. 4,626,288 which is  
incorporated herein by reference. In a particularly useful embodiment, the starch  
is derivatized by reaction with an alkenyl cyclic dicarboxylic acid anhydride by the  
method disclosed in U.S. Patent Nos. 2,613,206 and 2,661,349, incorporated  
10   herein by reference, or propylene oxide, more particularly by reaction with  
octenylsuccinic anhydride.

          In choosing a suitable starch, one skilled in the art would look to its ability  
to absorb and carry the chemical without leakage. This is important as the  
chemical must arrive at its destination and not be lost along the way. For  
15   example, when an oil well chemical is used, the starch must carry the chemical  
along the pipe or umbilical until it reaches the oil well. Thus, the starch must be  
stable in a transport medium, such as an organic, aqueous, or MEG medium  
used in the oil field industry.

          One skilled in the art would further look to the ability of the starch to  
20   release the chemical at its destination. Particulate starches effectively achieve  
this requirement. When the starch cooks out, it will release the adsorbed  
chemical. As different starches have different gelatinization temperatures, the  
starch may be chosen such that it will release its chemical at a selected  
temperature desired in the particular application.

25           The particulate starches of the present invention, including granular  
starch and resistant starch, may be combined with chemical reagents, oil well  
chemicals in particular, to form the controlled release chemical compositions of  
the present invention.



5 Oil well chemicals in accordance with this invention include, without limit, friction reducers, corrosion inhibitors, wax inhibitors, hydrate inhibitors, gel breakers, tracers, surfactants, scale inhibitors, antifoaming agents, demulsifiers, pour point depressants, biocides, drag reducers, antioxidants, hydrogen sulfide scavengers, oxygen scavengers, deoilers, and asphaltene inhibitors. Such  
10 chemicals can be in either liquid or solid form.

Scale inhibitors is intended to include without limitation phosphates, phosphate esters and inorganic phosphates, and (co-polymers of acrylates, maeates and sulphonates. Corrosion inhibitors is intended to include without limitation amines and polyamines; biocides such as aldehydes or  
15 halopropionamides. Oxygen scavengers is intended to include without limitation sodium bisulfite.

Other oil well reagents such as those disclosed in U.S. Patent No. 4,670,166, the disclosure of which is incorporated herein by reference, may also be suitable. Another category of oil well chemicals are the so-called breaker  
20 reagents which are introduced with fracturing fluids into subterranean formations to lower the viscosity of the fracturing fluid. Examples of such breaker reagents include without limitation sodium and ammonium persulfate, alpha and beta amylases and glucosideases and similar reagents such as disclosed in U.S. Patent No. 4,506,734, the disclosure of which is incorporated herein by reference.

25 Particularly suitable oil well chemicals are corrosion inhibitors, wax inhibitors, gas hydrate inhibitors, and asphaltene deposition inhibitors. Corrosion inhibitors include without limit, non-quaternized long aliphatic chain hydrocarbyl N-heterocyclic compounds and mono- or di-ethylenically unsaturated aliphatic

5 groups. Wax inhibitors include, without limit, polymers such polyethylene, or a copolymeric ester such as ethylene vinyl acetate copolymers, alpha olefin maleate, fumarate polyesters, and vinyl acetate polymers. Gas hydrate inhibitors may be any conventional hydrophilic hydrate inhibitor including, without limit, methanol, ethanol, hydroxy ethers, glycols, and ammonium chloride, sodium  
10 chloride or calcium chloride solutions. Asphaltene inhibitors include, without limit, amphoteric fatty acids or a salt of an alkyl succinate.

The controlled release chemical compositions of the present invention may optionally include additional adjuncts which improve controlled release or are themselves encapsulated by other encapsulation mediums known in the art.  
15 Possible adjuncts include, without limit, plasticizers, surfactants, fillers, chelating agents, thickening agents, particulates and other typical controlled release ingredients such as gelatin, gum arabic, zein, soy protein, and copolymer/polymers such as polyethylene and polyvinyl chloride.

Starch allows the compositions of the present invention to be made by a  
20 one-step adsorption process. The chemical compositions of the present invention may be prepared by mixing the desired oil well chemical with a starch particulate to form a mixture and stirring vigorously at ambient temperature and pressure. The amount of shear to which the starch/chemical mixture is exposed may need to be adjusted to ensure that the chemical is properly adsorbed onto  
25 the particulate starch.

Typically, the ratio of oil well chemical to starch is from about 30:70 to 80:20, more particularly from 40:60 to 60:40. Where practicable, the oil field chemical is first melted before adding the chemical to the starch particulate,

5 especially if it is a wax inhibitor that is solid at room temperature. Optionally, at least one encapsulation adjunct and/or solvent may be included in the formulation mixture. Further, weighting agents may be added to minimize gravity separation of the composition with the oil well medium.

The formulation mixture may also be optionally subjected to further  
10 treatment so that a stable, free-flowing powder having controlled release properties is obtained. After obtaining the free-flowing powder, the encapsulated chemical composition is then optionally milled to a desirable particle size diameter, preferably less than 20 microns. The particle size of the present invention should be large enough to inhibit inhalation and to prevent powdering  
15 when handling.

Optional treatments for obtaining the controlled release chemical compositions of the present invention include, without limit, subjecting the formulation mixture to treatments such as vacuum batch thermal processing, roll compaction, fluid bed processing, spray drying and extrusion. These processing  
20 treatments are well known to one of ordinary skill in the art. The processing conditions of the treatment should be limited to those that produce a chemical composition in which the starch retains its particulate structure.

The resultant chemical compositions are in the form of a biodegradable, non-sticky, free flowing powder having controlled release properties. Upon  
25 release of the adsorbed chemical, the starch will be carried by the aqueous phase and discharged into the environment. In contrast, the use of a solubilized starch in the formulation mixtures produces a sticky mixture that is unsuitable for controlled release applications.

- 5            Selection of the starch controls the release profile. For example, a starch which breaks down more easily or under less severe conditions will release the starch more quickly. Typically, the uncooked starch breaks down at high temperature, and thus is released when introduced to an aqueous or organic carrier fluid, forcing the expulsion of the encapsulated material into the stream.
- 10          However, the starch may be formulated to release under other conditions to which it would be exposed.

- The present compositions protect then adsorbed material from that of the medium, e.g. oilwell fluids, with which it may not be compatible and/or soluble. The starch allows for two incompatible (or compatible) oil well chemicals to be
- 15          carried in a medium. The starch also enables a higher loading of oil well chemical down in one injection line than without the starch and allows potentially hazardous materials to be handled easily and safely, limiting exposure. Further, the adsorption may make it possible to use materials that would otherwise have a flashpoint too low to be able to be used offshore.

- 20          The free flowing powder has preferable handling characteristics to the liquids when being used offshore. The dispersion of granular starch will be much easier than a fine powder if there is a high surface tension, or phase boundary, between the carrier starch and the oil well medium and is robust enough to be able to stand the shear stresses that will be employed to mix the material in the
- 25          carrying medium. i.e. impellers.

            The following examples are presented to further illustrate and explain the present invention and should not be taken as limiting in any regard.

5      **EXAMPLES**

          The following oil field chemicals were used:

- Corrtreat 2001-29, a solid, water soluble, amphoteric, betain-based surfactant for use as a corrosion inhibitor, commercially available from TR Oil Services
- Corrtreat 2001-30, a solid, trimercaptotriazine-based corrosion inhibitor, commercially available from TR Oil Services
- Hytreat 569, ethanediol 2-butoxyethanol propan-2-ol PV caprolactam for use as a kinetic hydrate inhibitor, commercially available from TR Oil Services
- Hytreat A560, quarternary ammonium 2-butoxyethanol diisobutyl ketone for use as an anti-agglomerate hydrate inhibitor, commercially available from TR Oil Services
- Scaletreat 2001-26, a solid, phosphate-based, water soluble scale inhibitor, commercially available from TR Oil Services,
- Scaletreat 2001-28, a solid, polymer-based, water soluble scale and corrosion inhibitor, commercially available from TR Oil Services
- Scavtreat 1020, a liquid, water miscible, triazine hydrogen sulphide scavenger, commercially available from TR Oil Services
- Trosquat, a liquid biocide formulation, commercially available from TR Oil Services
- Waxtreat 398 - a liquid, EVA wax inhibitor, commercially available from TROil Services
- Waxtreat 7302 - a liquid, resin-based asphaltene/wax dispersant, commercially available from TR Oil Services

Example 1:

- This example illustrates a method of preparing the biodegradable controlled release chemical compositions of the present invention.
- A granular starch (150 g, starch octenylsuccinate, aluminum salt, commercially available from National Starch and Chemical Company) was added to a pour point suppressant, XPC 3147C (50 g, Aldrich), which had been melted at a temperature greater than 30°C. The mixture was stirred at ambient

5 temperature and pressure in a high shear disperser (Torrence, #785049) at 2000-4000 rpm. An additional 100 g of the granular starch was added to the mixture and stirred for two more minutes to form a fine, free-flowing powder.

#### Example 2:

10 This example illustrates that the chemical compositions of the present invention comprising a particulate starch are superior to chemical compositions prepared with a solubilized starch.

Solubilized starch (250 g of a beta-amylase treated, 3% octenyl succinic anhydride modified waxy corn starch) was added to XPC 3147C (50 g, Aldrich) 15 which had been melted at a temperature greater than 30°C. The mixture was stirred at ambient room temperature and pressure in a high shear disperser (Torrence) at 2000-4000 rpm.

The resultant solid was sticky to the touch and, unlike the superior free-flowing powder of Example 1, was unsatisfactory for use as a controlled release 20 chemical composition.

#### Example 3.

This example illustrates compositions of the present invention using 25 water soluble oil well chemicals.

Water soluble solids were formulated with starch at a 1:1 ratio (50% loading on starch). The oil well chemical was solubilized in ambient water and homogenized for 1-2 minutes at 9000-10000 rpm (Silverson L4RT). The starch was then added to the solution and the mixture was further homogenized for 2-3 30 minutes at 9000-10000rpm, 20°C (Silverson L4RT). The mixture was spray dried (40% solids, 375°F inlet temperature, 225°F outlet temperature with a feed rate of 160ml/minutes and dual wheel atomization using Bowen Lab Model (30" x 36") to produce a flowable, non-sticky composition.

- 5 a. The example was carried out using a scale inhibitor, Scaletreat 2001-28, as the oil well chemical and Vulca 90, a maize starch crosslinked with 1.5% epichlorohydrin on dry starch.
- b. The example was carried out using a corrosion inhibitor, Corrtreat 2001-29 as the oil well chemical and a starch acetate (1.5 DS) waxy maize starch.
- 10 c. The example was carried out using a scale inhibitor, Scaletreat 2001-26 as the oil well chemical and a microporous waxy maize starch which was digested using 0.3% glucoamylase on dry starch to achieve 15% digestion.

Example 4.

- 15 This example illustrates compositions of the present invention using water insoluble oil well chemicals.

Water insoluble solids were formulated with starch at a 1:1 ratio (50% loading on starch). The oil well chemical was added to a waxy maize starch modified with 3% octenyl succinic anhydride and converted to a water fluidity of 40, and the mixture was homogenized for 1-2 minutes at 9000-10000 rpm, 20°C (Silverson L4RT). Water was added to the emulsion and the mixture was further homogenized, 1 minute at 9000-10000 rpm, 20°C (Silverson L4RT). The starch was then added to the solution and the mixture was further homogenized, 1-2 minutes at 9000-10000 rpm, 20°C (Silverson L4RT). The mixture was spray dried (35% solids, 380°F inlet temperature, 230°F outlet temperature, 140-160ml/minutes with dual wheel atomization using Bowen Lab Model (30" x 36")) to produce a flowable, non-sticky composition.

- 30 a. The example was carried out using a wax inhibitor, Waxtreat 398 as the oil well chemical and a microporous waxy maize which was 30% digested with 0.3% glucoamylase, and modified with 3% octenyl succinic anhydride and crosslinked with 1% aluminium sulfate.

- 5      b.      The example was carried out using an asphaltene inhibitor, Waxtrear  
7302 as the oil well chemical and a microporous waxy maize starch modified  
using 3% octenyl succinic anhydride, enzymatically treated using 0.3%  
glucoamylase, to achieve 30% digestion.
- 10      c.      The example was carried out using hydrogen sulphide, Scavtrear 1020 as the  
oil well chemical and a high amylose corn starch, HYLON® VII starch,  
commercially available from National Starch and Chemical Company.
- 15      d.      The example was carried out using a kinetic hydrate inhibitor, Hytrear 569 as  
the oil well chemical and a microporous (30% enzyme digested) waxy maize  
starch modified using 3% octenyl succinic anhydride, enzymatically treated using  
0.3% glucoamylase.
- 15      e.      The example was carried out using an anti-agglomerated hydrate inhibitor,  
Hytrear A560 as the oil well chemical and a cationic starch silanol, 0.3% Nitrogen,  
0.4% silanol.

- 20      Example 5 – This example shows further compositions representative of the  
present invention.

25      Starch was weighed out into a glass container. The oil well chemical was added  
while mixing for 5 minutes and then mixed for an additional 5 minutes, or until  
uniform using a Powerstat, Variable Autotransformer set at 80 (3PN168), Bodine  
Electric Co, Speed reducer motor (NSE-12R).

- 30      a.      Starch used was a 50:50 blend of sago and tapioca, DD and the oil well  
chemical used was Waxtrear 398. The starch:chemical ratio used was  
100:40 and the loading was 28.6%.
- 30      b.      Starch used was a high amylose (70%) maize starch modified by 3% octenyl  
succinic anhydride and 10% polyvinyl alcohol and the oil well chemical used  
was Waxtrear 398. The starch:chemical ratio used was 100:80 and the  
loading was 44.4%.



- 5 c. Starch used was enzyme converted (alpha amylase) maltodextrin and the oil well chemical used was Trosquat. The starch:chemical ratio used was 100:38 and the loading was 27.5%.
- d. Starch used was enzyme converted (alpha amylase) maltodextrin and the oil well chemical used was Troscat. The starch:chemical ratio used was 100:38 and the loading was 27.5%.
- 10 e. Starch used was a high amylose (70%) maize which was gelatinized, completely enzymatically debranched and retrograded and the oil well chemical used was Hytreat A560. The starch:chemical ratio used was 100:24 and the loading was 19.3.
- 15

While the invention has been described with particular reference to certain embodiments thereof, it will be understood that changes and modifications may be made by those of ordinary skill within the scope and spirit of the following claims.